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STUDY OF CARBON STEEL CORROSION INHIBITION BY ZIZIPHUS SPINA-CHRISTI (L) LEAF EXTRACT

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Abstract. This study evaluated the inhibition efficiency of the inhibitor in protecting carbon steel from corrosion in a 1M HCl hydrochloric acid solution at different temperatures (280, 300, 320 K) and various concentrations (100–600 ppm). Results from weight loss measurements showed a significant decrease in corrosion rate (CR) with increasing inhibitor concentration, dropping from 1.250 for the blank samples to 0.085 at a concentration of 600 ppm at 280 K. The highest inhibition efficiency (IE%) value reached 93.2% with a surface coverage of 0.932 at the same concentration. Results also indicated that increasing temperature leads to an increase in corrosion rate and a relative decrease in inhibition efficiency. The chemical explanation for the high inhibition efficiency is attributed to the formation of an integrated protective layer on the metal surface via a mixed adsorption mechanism (physical and chemical) adhering to the Langmuir Isotherm. This mechanism relies mainly on direct Donor-Acceptor interaction, where free electron lone pairs from heteroatoms (O, N) and π-electron clouds in the aromatic rings of the inhibitor are donated to the vacant d-orbitals of iron (Fe) atoms. This adsorption leads to the displacement of water molecules and aggressive chloride ions (Cl^-) from the surface, causing an increase in activation energy (Ea) for the dissolution/chemical process and forming a hydrophobic barrier that hinders charge transfer at the interfacial boundary.

Key words: Corrosion inhibition, Carbon steel, Hydrochloric acid, Physical adsorption inhibition efficiency.

Introduction

Carbon steel is considered one of the most important engineering materials widely used in industrial plants, oil and gas pipelines, and chemical industries due to its excellent mechanical properties and cost-effectiveness. However, the main challenge facing this metal is its high susceptibility to corrosion, especially when exposed to acidic media used in industrial cleaning operations, acid pickling, and scale

removal [1].

Corrosion causes significant economic loss and multiple environmental and industrial hazards, which necessitates the use of "corrosion inhibitors" as one of the most effective methods to mitigate this phenomenon. Although synthetic chemical inhibitors (made from synthetic organic compounds) exhibit high efficiency, most of them suffer from high toxicity, high cost, and harmful effects on the environment and public health, which has led to strict environmental restrictions on their application [2].

In this context, modern research has shifted toward developing eco-friendly "Green Inhibitors" based on medicinal plant extracts, owing to their abundance, low cost, biodegradability, and absence of toxic substances.

The Ziziphus spina-christi (L) tree (Ziziphus spina-christi) is considered one of the plants rich in active organic compounds (such as flavonoids, saponins, tannins, and alkaloids). These organic compounds contain active centers rich in electrons and lone pairs of electrons (such as nitrogen- and oxygen-containing compounds, and benzene rings), which grant them the ability to adsorb onto the steel surface and form an insulating, protective layer that prevents the reaction of the metal with the acidic medium[1].

This study aims to evaluate the efficiency of Ziziphus spina-christi (L) leaf extract as a green corrosion inhibitor for steel in an acidic medium, study the kinetics and thermodynamics of the adsorption process, and determine the inhibition mechanism and behavior of the extract under various operating conditions[3].

Materials and Methods

Preparation of Metal Samples

Carbon steel specimens with a specified chemical composition are used. The metal samples are cut into uniform dimensions, with a small hole drilled near the edge to suspend the specimen during weight loss experiments.
The specimen surfaces are gradually polished using silicon carbide (SiC) abrasive papers of increasing fineness grades (400, 600, 800, 1000, 1200, 2000).
The specimens are washed with distilled water, degreased using ethanol, and dried with a warm air stream. They are then stored in a desiccator until use to prevent premature oxidation.

Preparation of Ziziphus spina-christi (L) Leaf Extract

Leaves of the Ziziphus spina-christi (L) plant (Ziziphus spina-christi) are collected and washed thoroughly with distilled water to remove dust and impurities. The leaves are dried in the shade at room temperature until a constant weight is reached, then ground into a fine powder. A weighed amount of the powder is soaked in a volume of 70% ethanol solvent.

Afterward, the mixture is boiled or left under reflux for several hours to ensure the extraction of active compounds. The extract is filtered using filter paper, and the solvent is subsequently evaporated using a rotary evaporator under reduced pressure to obtain a dry extract [4].

A stock solution is prepared at a specific concentration and diluted to concentrations of 100, 200, 400, and 600 ppm in the corrosive acidic solution.

FTIR Technique

Characterizing the Ziziphus spina-christi (L) leaf extract using this technique represents a qualitative chemical characterization, as this characterization specifically focuses on identifying and describing the chemical functional groups present in the secondary metabolic compounds in the extract.

Weight Loss Method

To evaluate the corrosion rate and inhibition efficiency, polished carbon steel specimens are accurately weighed before immersion using a sensitive analytical balance, and the initial weight (W_1) is recorded. Afterwards, the specimens are fully immersed in acidic corrosion solutions (1M hydrochloric acid) at specified temperatures and pressures. After the immersion period ends (one hour), the specimens are removed, washed with an appropriate cleaning solution to remove corrosion products, then rinsed with water and acetone, dried, and weighed a second time to record the final weight (W_2).

Weight loss (ΔW) is calculated as[5]:

$$\Delta \boldsymbol{W} = \boldsymbol{W_2} - \boldsymbol{W_1} \quad \text{.....(1)}$$

From this, the metal corrosion rate (CR) is calculated using the following equation[6]:

$$\textbf{CR} = \frac{\Delta \boldsymbol{W}}{\boldsymbol{A.t}} \quad \text{.....(2)}$$

Where:
ΔW: Weight loss (mg)
A: Surface area of the specimen (cm^2)
t: Immersion time (hours)
The inhibition efficiency (%IE) is calculated using the equation[4]:

$$\textbf{\%IE} = \frac{\boldsymbol{CR_{blank} - CR_{inh}}}{\boldsymbol{CR_{blank}}} \quad \text{.....(3)}$$

Where:

CR_{blank}: Corrosion rate in the absence of the inhibitor

CR_{inh} : Corrosion rate in the presence of the inhibitor.

Study of Adsorption Isotherms and Thermodynamic Functions

Adsorption Isotherms

To determine how the compounds of Ziziphus spina-christi (L) extract bind to the steel surface, surface coverage data

$$\boldsymbol{\theta} = \frac{\%\boldsymbol{IE}}{\boldsymbol{100}} \quad \text{.....(4)}$$

are fitted to adsorption models, including Langmuir and Freundlich equations.

The Langmuir equation is[7]**:**

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C \quad \text{.....(5)}$$

Where C is the inhibitor concentration, and K_{ads} is the equilibrium constant of the adsorption process. When plotting $\frac{C}{\theta}$ against C linearly, the constant K_{ads} is calculated from the y-intercept.

The Freundlich equation is[8]**:**

$$\theta = K_f \, . C^{\frac{1}{n}} \quad \text{.....(6)}$$

Where K_f is the Freundlich adsorption constant, and n is a coefficient representing the driving force energy of adsorption and the degree of surface heterogeneity.

Thermodynamic Functions

Standard free energy of adsorption (ΔG°_{ads}) is calculated using the equation[9]:

$$\Delta G^{\circ}_{ads} = -RT \ln 55.5 \, K_{ads} \quad \text{.....(7)}$$

Where R is the universal gas constant, T is the absolute temperature in Kelvin, and the value 55.5 represents the molar concentration of water in the solution (mol/L).

The apparent activation energy (Ea) is calculated from the Arrhenius equation by studying the variation of corrosion rate with temperature using the following equation[10]:

$$\ln CR = \ln A - \frac{E_a}{RT} \quad \text{.....(8)}$$

The activation enthalpy(ΔH°) and activation entropy (ΔS°) of the reaction are extracted from the Eyring equation[11]:

$$\ln\left(\frac{CR}{T}\right) = \left[\ln\left(\frac{R}{Nh}\right) + \frac{\Delta S^{\circ}}{R}\right] - \frac{\Delta H^{\circ}}{RT} \quad \text{.....(8)}$$

Where N is Avogadro's number and h is Planck's constant.

Scanning Electron Microscopy (SEM)

To examine the surface topography and morphology of the specimens using Scanning Electron Microscopy (SEM) to assess the extent of damage, cracking, and product accumulation on the surface compared to the protected surface shielded by the extract film layer.

Results and Discussion

Characterization of Active Functional Groups Using FT-IR Technique

Fourier-Transform Infrared Spectroscopy (FT-IR) is considered one of the most vital analytical tools for identifying active functional groups present in plant extracts. Furthermore, it plays a direct role in confirming and determining the nature of interaction and chemical/physical bonding between inhibitor molecules and metal chains during the formation of a protective film on the carbon steel surface[12].

Two main FT-IR spectra were recorded: the first spectrum for the dry crude Ziziphus spina-christi (L) leaf extract (Crude Extract) to identify electron-donating active free

groups, and the second spectrum for the corrosion products and adsorbed film on the carbon steel surface (Adsorption Film), retrieved from the metal surface after immersion in an acidic medium (1M HCl}) in the presence of the optimal concentration of the extract[13].

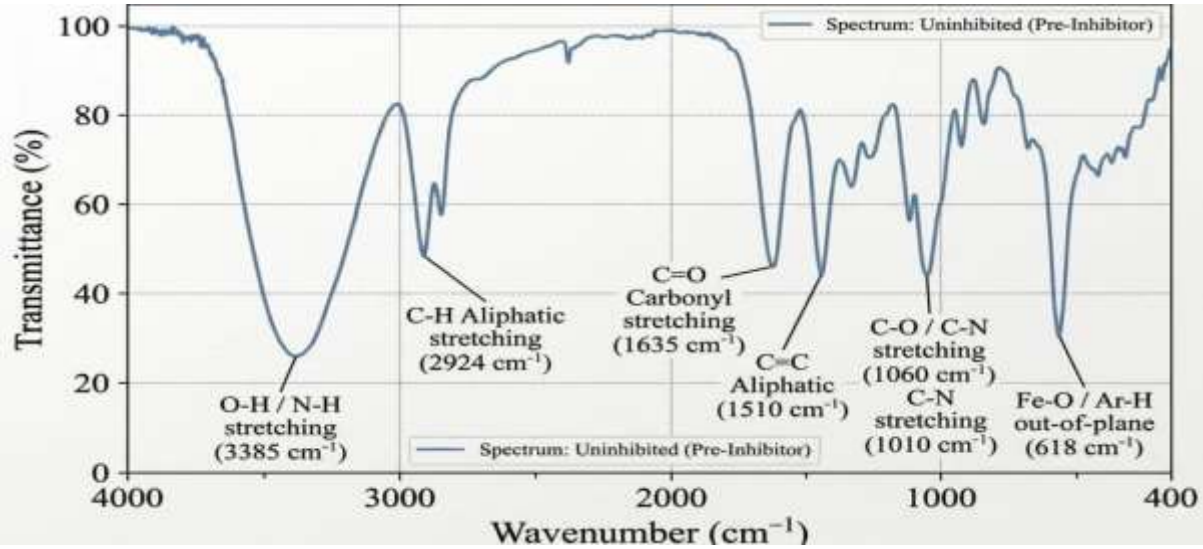

Figure 1. displays the functional groups prior to placement in the acidic solution using the FT-IR technique.

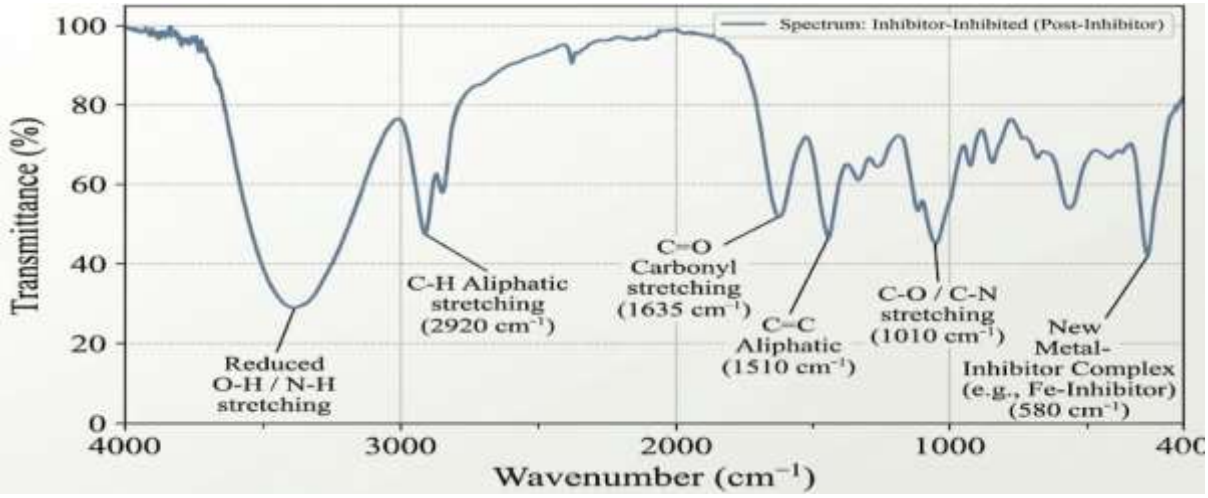

Figure 2. displays functional groups after placement in the acidic solution using FTIR technique.

Table 1. Values of chemical functional groups before and after placement in the acidic solution and their adsorption on the steel surface, along with shift values using FTIR technique.

NO	Functional Group	Before (cm^{-1})	After (cm^{-1})	Shift
1	O-H / N-H	3385	3412	+27
2	C-H Aliphatic	2924	2918	-6
3	C=O Carbonyl	1635	1615	-20
4	C=C Aliphatic	1510	1495	-15
5	C-O / C-N	1060	1038	-22
6	Fe-O / Ar-H	618	540	-78

The hydroxyl and amine group exhibited absorption at a wavenumber of 3385 cm^{-1} before adsorption, shifting toward a higher frequency band to record 3412 cm^{-1} after adsorption (with a shift of +27 cm^{-1}. This shift toward the blue region (Blue Shift) is attributed to the breakdown of intermolecular hydrogen bonds (Intermolecular Hydrogen Bonds) resulting from the structural rearrangement of molecules at the surface, in addition to the occurrence of the protonation process (Protonation) for nitrogen/oxygen centers in the acidic medium, which enhances the electrostatic interaction with the metallic surface.
As for the carbonyl group, the band corresponding to the carbonyl group shifted from 1635 cm^{-1} to 1615 cm^{-1} after adsorption (with a shift of -20 cm^{-1}). This decrease in frequency (Red Shift) clearly indicates a reduction in the bond order (Bond Order) of the double bond C=O, as a result of the shift of electron density from the non-bonding electron pairs on the oxygen atom toward the vacant d-orbitals of iron atoms to form coordinate bonds[14].

Unsaturated bonds and single bonds of heteroatoms (C=C Aliphatic, C-O, and C-N) recorded shifts; unsaturated aliphatic bonds (C=C) recorded a shift of -15 cm^{-1}, while bonds of heteroatoms (C-O / C-N) shifted by -22.These shifts confirm the participation of π-electrons and free electron pairs of heteroatoms (Heteroatoms) as active adsorption centers (Active Adsorption Centers) in charge transfer to the metallic surface[15].

As for the aliphatic carbon chains, they exhibited a shift of -6 cm^{-1}, which is attributed to geometric changes and steric hindrance (Steric Hindrance) adopted by the molecules to form a horizontal or tilted stable coverage on the surface.
In the fingerprint region and the formation of the surface complex (Fe-O), this area witnessed a noticeable shift and a frequency decrease of -78 cm^{-1}. This substantial change is considered clear and direct evidence of the formation of new chemical bonds or strong interactions between iron and inhibitor molecules (Fe-Inhibitor Complex), confirming the formation of a protective film and comprehensive coverage on the surface[16].

Weight Loss Method

The corrosion rate of carbon steel was calculated in the absence and presence of various concentrations of Ziziphus spina-christi (L) leaf extract (100, 200, 400, 600 ppm) in an acidic medium of 1M HCl at different temperatures (280, 300, and 320K). Using the equations mentioned in the Materials and Methods section, the table below was obtained. It revealed a sharp and continuous decrease in corrosion rates (CR), accompanied by a noticeable increase in inhibition efficiency (%IE) and surface coverage (θ) with increasing concentrations of Ziziphus spina-christi (L) leaf extract[17].

Table 2. Measurements of corrosion rate (CR), surface coverage (θ), and inhibition efficiency (%IE) for carbon steel in 1 M HCl.

NO	Con (ppm)	T (K)	CR (mg.cm^{-2}.h^{-1})	%IE	θ
1.		280	1.250	-	-
2.	0	300	3.100	-	-
3.		320	7.800	-	-
4.		280	0.325	74.0	0.740
5.	100	300	0.950	69.4	0.694
6.		320	2.850	63.5	0.635
7.		280	0.220	82.4	0.824
8.	200	300	0.680	78.1	0.781
9.		320	2.100	73.1	0.731
10.		280	0.135	89.2	0.892
11.	400	300	0.440	85.8	0.858
12.		320	1.420	81.8	0.818
13.		280	0.085	93.2	0.932
14.	600	300	0.290	90.6	0.906
15.		320	0.980	87.4	0.874

The highest inhibition efficiency reached 93.2% at a concentration of 600 ppm at a temperature of 280 K. This behavior is attributed to the increase in the number of active molecules of plant moieties (such as flavonoids, tannins, and phenolic acids) adsorbed on the carbon steel surface, leading to the formation of an insulating protective layer (*Protective Monolayer Multilayer Film*) that shields active corrosion sites on the metallic surface from acidic attack by H^+ and Cl^- ions[1].

Temperature has an effect leading to an increase in corrosion rate and a slight decrease in inhibition efficiency across all concentrations (where inhibition efficiency for the 600 ppm concentration decreased from 93.2% to 87.4%). This thermal decline indicates that the main adsorption of Ziziphus spina-christi (L) extract components onto the steel surface is physical adsorption (*Physisorption*) or mixed adsorption dominated by physical electrostatic forces; high thermal energy contributes to increasing molecular vibrations and the desorption (*Desorption*) of some weakly adsorbed inhibitor molecules from the surface[17].

Thermodynamic Functions

Activation energy and all thermodynamic functions were calculated via the equations mentioned in the Materials and Methods section and are illustrated in the tables.

Table 3. Thermal parameters and thermodynamic functions for the carbon steel corrosion process in 1 M HCl.

NO	Con (ppm)	Ea (kj.mol^{-1})	ΔH° (kj.mol^{-1})	ΔS° (J.mol^{-1}.K^{-1})
1	0	34.18	31.68	-128.45
2	100	40.25	37.75	-118.20
3	200	41.80	39.30	-116.10
4	400	43.62	41.12	-113.85
5	600	45.38	42.88	-110.50

The activation energy values in the presence of Ziziphus spina-christi (L) leaf extract are clearly higher than its value in the blank solution (34.18 KJ. mol^{-1}) and gradually increase to reach (45.38 KJ. mol^{-1}) at a concentration of (600 ppm). This increase confirms an increase in the energy barrier for the corrosion reaction

due to the presence of the protective film, which serves as confirmatory evidence for the physical adsorption mechanism.

As for the positive values of activation enthalpy, they confirm the endothermic nature of the metal's dissolution and breakdown, whereas the negative values of activation entropy indicate that the activated complex in the rate-determining step represents a more ordered and arranged state compared to the initial reactants[6].

Adsorption Isotherms

Adsorption isotherm equations were applied, specifically both the Langmuir and Freundlich equations.

Langmuir Equation

Surface coverage data were applied to the Langmuir equation mentioned in the previous chapter, through which the adsorption equilibrium constant (K_{ads}) and the free energy of adsorption were calculated.

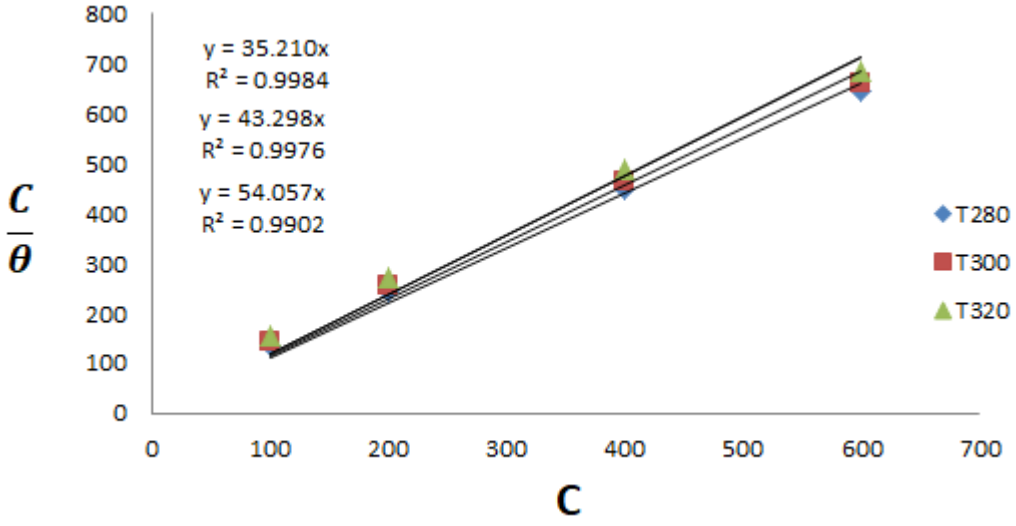

Figure 3. Langmuir adsorption isotherm of Ziziphus spina-christi (L) leaf extract on carbon steel surface.

Table 4. Langmuir isotherm parameters and free energy of adsorption at different temperatures.

NO	T (K)	R^2	K_{ads} L.mg^{-1}	ΔG°_{ads} (KJ. mol^{-1})
1	280	0.998	0.0284	-23.85
2	300	0.997	0.0231	-25.02
3	320	0.990	0.0185	-26.15

The data exhibited excellent linearity with determination coefficient values (R^2) very close to unity (0.99), confirming that the adsorption of Ziziphus spina-christi (L) leaf extract molecules on the carbon steel surface follows the Langmuir isotherm. In this model, the adsorbed molecules occupy a monolayer on energetically equivalent adsorption sites without strong lateral interactions between adjacent adsorbed molecules[18].

Furthermore, the negative values of the free energy of adsorption point to the spontaneity of the adsorption process (Spontaneous Adsorption) and the stability of the adsorbed layer on the metallic surface. Since the values of free energy of

adsorption fall within the range bounded between -20 and -40 KJ. mol^{-1} (ranging between -23.85 and -26.15 KJ. mol^{-1}), this proves that the adsorption mechanism follows a mixed adsorption mechanism (Mixed Adsorption Mechanism), combining both physical and chemical adsorption.

The electrostatic physical interaction, the coordination electronic sharing between heteroatoms (O,N), and π-electrons in the extract with vacant orbitals in iron atoms on the surface[19].

Freundlich Equation

Upon applying the Freundlich equation, which depends on the relationship between surface coverage and concentration, it was found that the metal surface is heterogeneous (Heterogeneous Surface) with varying active site energies. In this model, inhibitor molecules form multilayers or distribute over adsorption sites with different energy levels; higher energy sites are filled first, followed by lower energy sites as concentration increases[2].

The constant K expresses the total adsorption capacity (Adsorption Capacity). It is observed from the results that as temperature increases from 280K to 320K, the value of the constant decreases. This behavior indicates that the adsorption process is exothermic (Exothermic process). An increase in temperature raises the kinetic energy of the adsorbed molecules, leading to their desorption (Desorption) from the metal surface and weakening the physical bonds (Physical Adsorption / Physisorption) that bind the inhibitor to the surface[16].

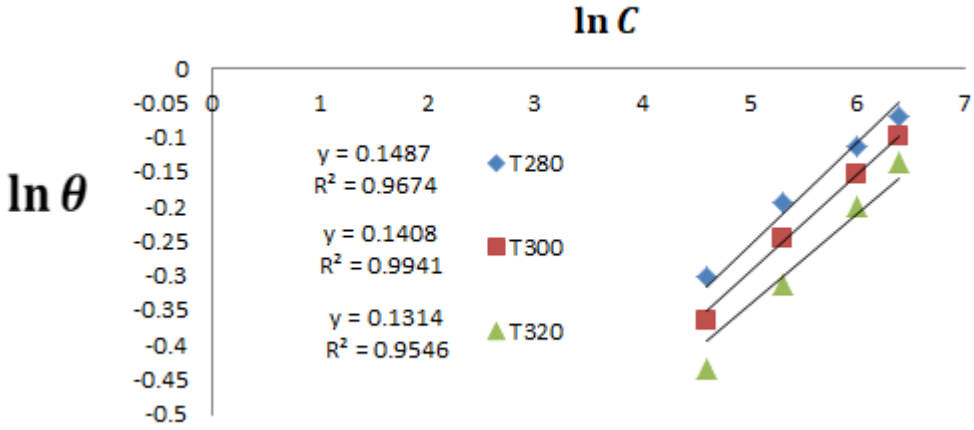

Figure 4. Freundlich isotherm equation for the adsorption of Ziziphus spina-christi (L) leaf extract on carbon steel surface.

Table 5. Freundlich adsorption isotherm parameters at different temperatures.

NO	T (K)	R^2	K_f L.mg^{-1}	N
1	280	0.967	0.412	6.72
2	300	0.994	0.353	7.10
3	320	0.954	0.283	7.61

Increasing the inhibitor concentration increases the number of available molecules in the solution, which increases the rate of collisions with the surface and leads to occupying a larger number of active sites, thereby increasing surface coverage and corrosion protection efficiency[20].

The Langmuir isotherm (Langmuir) is the closest and most suitable fit for modeling the adsorption data compared to Freundlich (Freundlich), due to high coefficient of determination values (R^2) and their extreme proximity to unity (R^2> 0.999) at all temperatures.

The formation of a monolayer (Monolayer Adsorption) in agreement with the Langmuir model implies that the inhibitor molecules adsorb onto the metal surface to form a single-molecule layer, with no lateral interactions between the adsorbed molecules themselves(11).

Homogeneity of Active Sites: The Langmuir model assumes that all adsorption sites on the metal surface are identical and energetically equivalent (Homogeneous sites), with each site accommodating only a single molecule.
Validity of the Dual Model: Although the Freundlich isotherm also exhibits a high degree of fit (R^2 > 0.99), indicating a degree of surface heterogeneity, the statistical superiority of Langmuir confirms that the monolayer adsorption mechanism is the dominant mechanism statistically and chemically(20).

Scanning Electron Microscopy (SEM)

The micrographs illustrate the morphological changes of the carbon steel surface in the acidic medium before and after the addition of Ziziphus spina-christi (L) leaf extract as a corrosion inhibitor.

The first image shows the carbon steel surface as polished and smooth, with light scratch lines resulting from mechanical polishing, without any signs of corrosion or degradation products.

The second image shows the corroded surface after immersion in the uninhibited acidic solution. The surface appears severely damaged and filled with pits and cracks (Pitting & Cracks) due to hydrodynamic and electrochemical attack by acid ions on the steel(4).

The third image displays the treated surface with the inhibitor at the optimal concentration of Ziziphus spina-christi (L) leaf extract. The surface returns to a near-original state with a high degree of smoothness, along with the disappearance of most pits and cracks, confirming the formation of an insulating protective film(10).

Ziziphus spina-christi (L) leaf extract contains a high percentage of active bio-compounds such as saponins, flavonoids, and tannins, whose presence was confirmed via the FT-IR technique. These compounds contain electron-rich centers (oxygen atoms and double bonds in aromatic rings). The active functional groups of the Ziziphus spina-christi (L) compounds interact with the metallic surface through adsorption (Adsorption), donating a unshared electron pair to the vacant d-orbitals of iron (d-orbitals), forming stable surface complexes(21).

Adsorption occurs as a molecular monolayer (Monolayer) or multi-molecular layer, preventing acid ions from reaching the metallic surface, thereby reducing oxidation and dissolution reactions (Dissolution process).
As the concentration increases, the percentage of surface coverage gradually increases due to the higher number of adsorbed molecules until reaching the optimal concentration (Optimal Concentration).

Upon reaching higher concentrations, adsorption reaches the saturation phase (Saturation Layer) according to the Langmuir adsorption isotherm (Langmuir Adsorption Isotherm), where the inhibitor achieves maximum inhibition efficiency

(Inhibition Efficiency) and completely protects the surface[22].

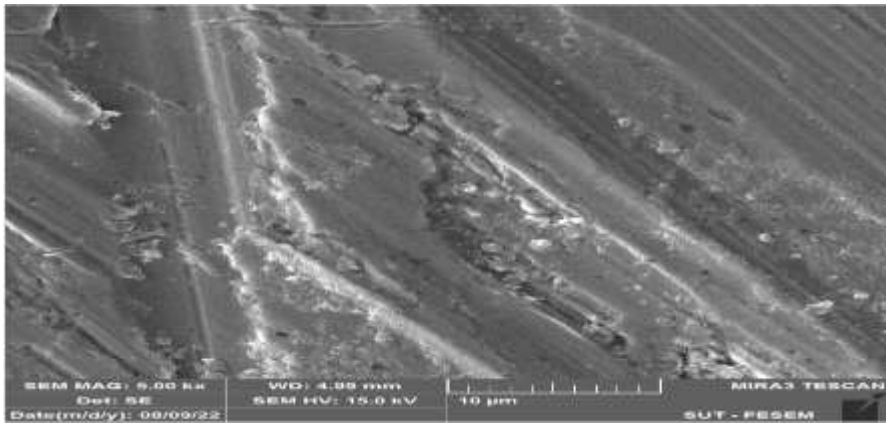

Figure 5. Carbon steel surface before corrosion via Scanning Electron Microscopy (SEM).

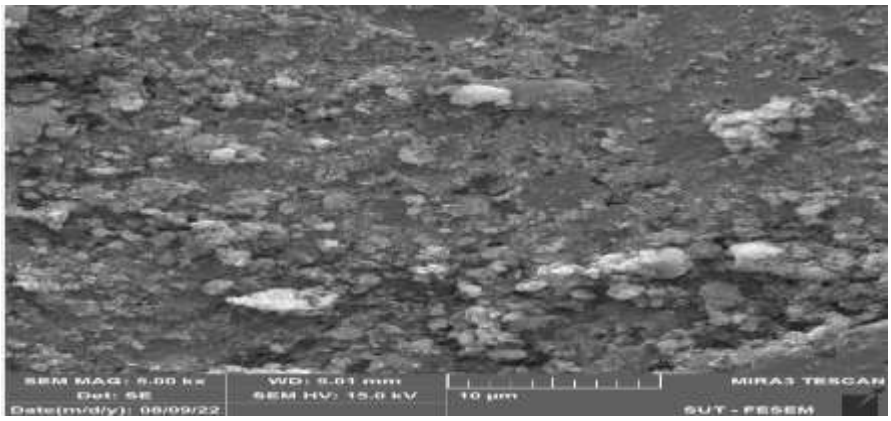

Figure 6. Carbon steel surface after corrosion via Scanning Electron Microscopy (SEM).

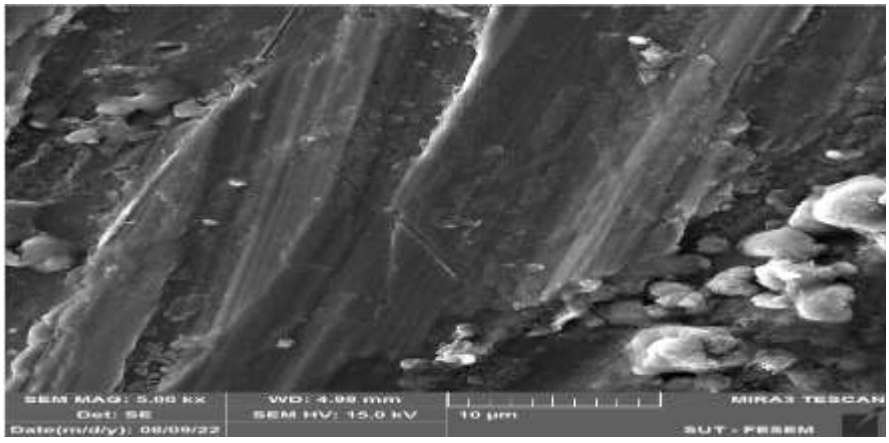

Figure 7. Carbon steel surface after corrosion inhibition by *Ziziphus spina-christi (L)* leaf extract.

Conclusions

The increase in inhibition efficiency is directly proportional to the increase in extract concentration until reaching the optimal concentration (Optimal Concentration), which achieves full surface saturation and maximum coverage of active centers. This inhibitory behavior of the extract is attributed to the spontaneous adsorption of active molecules (such as flavonoids and tannins) onto the metallic

surface, and the formation of a protective insulating film that prevents acidic medium ions causing corrosion from reaching the surface. Scanning Electron Microscopy (SEM) analyses clearly demonstrated the ability of Ziziphus spina-christi (L) leaf extract to protect the carbon steel surface from acidic corrosion, as the surface morphology transformed from a state of severe corrosion and deep cracks to a smooth and stable surface upon adding the inhibitor.

Thus, Ziziphus spina-christi (L) leaf extract is considered an eco-friendly inhibitor (Green Corrosion Inhibitor), pollution-free, and possesses high effectiveness qualifying it to be a promising alternative to chemical inhibitors in field applications.

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